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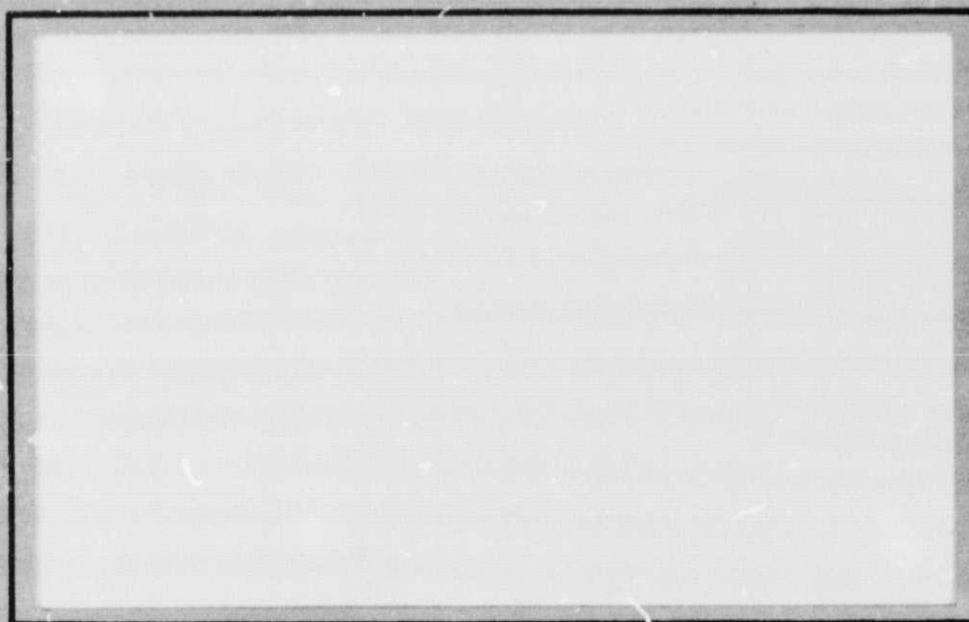
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March 1978

A NEW EXPERIMENTAL METHOD FOR THE ACCELERATED
CHARACTERIZATION OF COMPOSITE MATERIALS

Y. T. Yeow, Research Associate
D. H. Morris, Associate Professor
H. F. Brinson, Professor

Department of Engineering Science and Mechanics

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ABSTRACT

The use of composite materials for a variety of practical structural applications is presented and the need for an accelerated characterization procedure is assessed. A new experimental and analytical method is presented which allows the prediction of long-term properties from short-term tests. Some preliminary experimental results are presented.

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INTRODUCTION

Polymer matrix fiber reinforced materials have enormous potential for use in a wide variety of structural applications including automobiles, sporting equipment, airplanes, etc. In fact, graphite/epoxy tennis rackets, skis, and golf club shafts are already widely used. In an effort to reduce weight and conserve fuel, modern automobiles contain many parts manufactured from plastics and chopped and continuous glass polyester composites. Missiles and aircraft also currently use graphite/epoxy and many other types of laminated composites.

Probably the largest single inhibitor to even greater usage of polymer based composites is the current high cost. However, it is likely that prices will be substantially reduced in the future due to increased usage, new manufacturing developments and as increased fuel conservation measures demand strong but lightweight materials for structural applications.

In addition to cost, technical reasons exist which inhibit the ready acceptance of polymer-based composites for structural applications in automobiles, airplanes, etc. One such factor is the current lack of understanding of the mechanical behavior of polymer based laminates under long-term environmental exposure. It is well known that the epoxy resins which are now often used as the polymer matrix component exhibit viscoelastic or time effects which are significantly affected by exposure to both temperature and humidity. Epoxies soften as temperatures are increased with resulting loss of both moduli and strength [1-4]. In addition, they absorb moisture and swell giving

rise to residual stresses [5-8].

Obviously, polymer based composite laminates will be similarly affected by moisture and temperature under certain circumstances. Fiber-dominated composites are not likely to suffer large reductions of either moduli or strength in the fiber direction. In other directions, properties will be affected by losses of both strength and modulus. Perhaps more importantly, drastic interply effects will occur. That is, delaminations due to internal residual stresses caused by both temperature and humidity are likely to occur together with a general loss of interply shear transfer capacity.

Because of these effects of environment, there is concern that time-dependent properties such as creep, relaxation, delayed failures, creep ruptures, etc., may be important long-term design considerations for the temperature and moisture levels anticipated in current structural applications. Further, it would be extremely desirable to be able to measure these effects with short-term laboratory tests rather than perform long-term prototype studies. It would also be desirable to be able to predict these effects with analytical techniques for either short or long-term situations. As a result, it is clear that there is an urgent need for accelerated characterization techniques for laminates similar to those used for other structural materials.

The purpose of the current research was to investigate the effect of temperature on graphite/epoxy laminates and to explore ways and means of their accelerated characterization--both experimentally and analytically which would compliment previous efforts related to fracture [9-19].

BACKGROUND

As mentioned above, the purpose of the current effort was to study and measure the thermomechanical viscoelastic response of graphite/epoxy laminates and in so doing to develop methods for the accelerated characterization of moduli and strengths and/or lifetimes of a structural component. That such viscoelastic effects are important has been previously demonstrated [19]. This result is reproduced and shown in Figure 1 which indicates that a delayed viscoelastic fracture process was observed for a graphite/epoxy $[\pm 45^\circ]_{4s}$ tensile specimen containing a circular hole. That is, a creep to failure response occurred for individual plies and the laminate eventually fractured (separated) even though the applied laminate tensile load was relaxing in a fixed grip situation. Obviously, should the same phenomena occur in prototype structure, unsafe premature failures would occur.

To be able to predict the lifetime of a structure under known loadings requires either long-term testing or a model upon which long-term results can be predicted from short-term tests. Obviously, long-term testing equivalent to the duration of the lifetime of a structure is undesirable. The alternative is to develop analytical or experimental models which can be successfully used for extrapolation. For metals and polymers a variety of techniques are available such as, linear elastic stress analysis, empirical extrapolative equations such as the Larson-Miller parameter method, Minor's rule and frequency independence, and time-temperature superposition principle, etc.

Two procedures have been proposed for the purpose of making such lifetime or viscoelastic predictions of composite materials. These are the "wear out model" proposed by Halpin, et al. [20] and a non-linear viscoelastic technique proposed by Lou and Shapery [21]. The former is a method based upon statistical reliability concepts and the premise that all materials contain inherent flaws which grow to critical sizes under various conditions of stress, strain, time, temperature, etc. The method employs viscoelastic shift type parameters together with a power law crack growth model to make accelerated predictions of strength. The method requires a thorough knowledge of statistical methods and significant test programs for each structural component in order to obtain an adequate sample size.

Shapery's technique employs a single integral non-linear viscoelastic stress strain characterization equation based upon a modified superposition principle (MSP) of the Boltzman type. He uses creep and creep recovery tests to determine the necessary parameters at a single elevated temperature but at different stress levels. Shift factors are developed for the octahedral shear stress at various stress levels and for various fiber angles for the unidirectional glass/epoxy materials investigated. Master curves are developed for the non-linear creep compliance. The method is developed only for unidirectional laminates and is not necessarily applicable to other loading conditions, e.g., relaxation.

We have decided to take a different approach from the above two methods. Our efforts are being based upon the time-temperature superposition principle and the widely used lamination theory for composite

materials.

In recent years the concept of the time-temperature superposition principle has been extended for the case of elastomers to include failure or fracture processes as well as the familiar moduli master curve concept [22,23]. In fact, Landel speaks of a stress-strain-time-temperature response surface. He argues rather convincingly that one need not determine the entire surface, but merely a portion of the surface. That is, by finding a trace of the surface, the remaining portions can be found. Whether similar reasoning can be used for composites, or even glassy polymers, remains to be seen. Lohr did show that the time-temperature superposition principle could be used for the yield strength of certain glassy polymers and that the shift function for strength was similar to that for modulus [24].

ACCELERATED CHARACTERIZATION PLAN

The procedures we are using for accelerated characterization and lifetime predictions are outlined in Figs. 2 and 3. As shown in Fig. 2, our plan is to determine the modulus master curve for unidirectional G/E laminates from short-term (15 min.) tensile creep tests from room temperature (20°C) to about 30°C above the T_g (180°C). This is to be accomplished for various fiber angles from 0° to 90° with respect to the load direction. From this series of tests shift functions vs. temperature and fiber angle are to be determined as indicated in Fig. 2b. Next, the assumption of linear viscoelasticity will be evoked and the lamina tensile and shear strength master curve will be obtained. That is, the strength master curve will be assumed to have the same

character as the modulus master curve. Initial values of strength will be needed to quantify the strength scale and will be taken from previous work [18].

Probably the most important aspect of the plan will be the generation of a shift function equation or relation of the WLF type as shown in Fig. 2d, which will be valid for the particular composite investigated, i.e., T300/934. If such is possible, then the remaining portion of the plan shown in Fig. 3 should follow and be reasonably accurate.

The procedure shown in Fig. 2 involves substantially more testing than that desirable for the usual design process. In other words, it would be desirable to base design on a minimum number of tests and probably only on those necessary for ordinary quality control. We envision the final accelerated design process as illustrated by the flow chart shown in Fig. 3. The first step in the process for any particular laminate would be to determine the modulus and strength design variables. Next, sufficient testing would be accomplished to produce a $[90^\circ]$ lamina master curve and to establish the necessary parameters for the shift function relation for that particular composite. With the shift function equation, master curves for moduli and strengths would be constructed. These master curves would be used as input to an incremental computational procedure using standard stress analysis lamination theory.

With the procedure outlined accelerated predictions of properties and lifetimes could be made for arbitrary laminates. Our intent is to make such calculations for matrix dominated laminates such as $[\pm\theta]_s$, $[90^\circ/\pm\theta/90^\circ]_s$, etc. Fiber dominated laminates will need similar calculations only for such situations as fiber matrix delaminations as these

laminates in general do not display normal time or rate dependent properties [15].

Once the laminate predictions are made, a testing program will obviously need to be instituted to verify those predictions.

PRELIMINARY RESULTS

Some of our preliminary findings are shown in Figs. 4-9. The thermal expansion measurements shown in Fig. 4 have established the dry glass-transition temperature of $T_g = 180^\circ \text{C}$ for our T300/934 G/E laminate. The same results have indicated a smaller secondary transition of $T_g^S = 60^\circ \text{C}$. In addition to these results, considerable effort was expended to determine the effect of thermal and mechanical cycling. We have learned that neither substantially alters the mechanical properties. Thus, no mechanical conditioning need be accomplished as Shapery found was necessary for glass-epoxy materials [21]. Further we established that repeated use of the same specimen could be made thus avoiding the additional tedium of adequate statistical sampling.

Figs. 5 and 6, respectively, show the unreduced one-minute reciprocal of compliance (modulus) vs. temperature results and the reduced 15-minute reciprocal of compliance (modulus) vs. time results for temperatures from $T = 20^\circ \text{C}$ to $T = 210^\circ \text{C}$ for our $[90^\circ]_{8S}$ laminate. In each case, the results are as expected and are quite similar to the results one would obtain for a neat epoxy resin [1,2]. Superposed on Fig. 6 is a portion of creep reciprocal of compliance (modulus) master curve for $T_0 = T_g = 180^\circ \text{C}$. The entire master curve for $T_0 = 180^\circ \text{C}$ is shown in Fig. 7 for the $[90^\circ]_{8S}$ material. As may be seen, the curve

stretches over about 22 decades of log time. Portions of the curve were obtained by extrapolating creep reciprocal of compliance vs. temperature data such as that shown in Fig. 5 and by using a polynomial representation of the data obtained with a computer program. This was necessary due to the small variations of properties at low temperature levels and was necessary in order to see the effect of the second order T_g .

Figure 8 shows the shift functions necessary to obtain a smooth master curve for $\frac{1}{S_{22}}$. As may be observed, the shift functions above T_g are approximated reasonably well by a WLF type equation although the constants are not the same as those used by Williams, Landel and Ferry and some deviations are noted.

Other master curves were generated for tensile specimens whose fibers were at an angle with respect to the load direction. Using an additional master curve for $\theta = 10^\circ$, the fact that no time or temperature effects were found for $\theta = 0^\circ$ (fiber dominated direction) and the visco-elastic analogue to the orthotropic transformation equation, the master curve for an arbitrary off-axis tensile specimen was predicted. These predicted results are shown in Fig. 9 for a specimen whose fiber to load angle was $\theta = 30^\circ$. Also shown is the master curve generated from short term test. As may be observed, the predicted master curve and experimentally determined master curve are in close agreement.

Twenty-five hour creep tests were performed on all geometries to verify both the process of using master curves as obtained from short-term (15 min.) tests and the analytical prediction procedures discussed above. These results are shown superimposed on the master curves given in Figs. 5 and 9. In the former, obviously discrepancies between the

short- and long-term results are obvious. On the other hand, close agreement between short-term, long-term and predicted results occurred for the latter case. It is felt that the deviations encountered are primarily due to the normally encountered statistical variations in the properties of composite materials and not due to the methods used.

SUMMARY AND CONCLUSIONS

A new method which allows the prediction of long-term properties of composite materials from short-term (15 min.) laboratory test results has been presented. Conceptually, the procedure outlined could be used for all types of laminates through modification of the standardly used lamination theory methods.

Experimental data has been presented which illustrate the nature of the viscoelastic process in graphite/epoxy materials. A delayed viscoelastic fracture was demonstrated to occur in a $[\pm 45^\circ]_{4s}$ laminate. Further, the time and temperature response of a matrix dominated uni-directional $[90^\circ]_{8s}$ laminate has been presented. It has been shown that master curves from short-term tests of $[90^\circ]_{8s}$ and $[30^\circ]_{8s}$ can be used to generate long-term master curves and that reasonable correlation can be obtained between the two results. Further, it has been shown that analytical predictions from short-term results can be successfully used to predict long-term (25-hour) results.

From the results presented herein, we have shown that the master curves and shift functions in Fig. 2 needed for our new accelerated characterization procedure outlined in Fig. 3 can be obtained and can be used with reasonable accuracy. Thus, we have shown that the

procedures of Fig. 3 for the long-term predictions of laminate properties, including strengths, is viable. Additional efforts are continuing to predict and measure the response of laminates over prolonged time periods, i.e., several weeks to several months.

ACKNOWLEDGEMENTS

The financial support provided for this work by NASA Grant NSG 2038 from the Materials and Physical Sciences Branch of NASA-Ames is gratefully acknowledged. Further, sincere appreciation is extended to H. G. Nelson and D. P. Williams for their encouragement and many helpful discussions.

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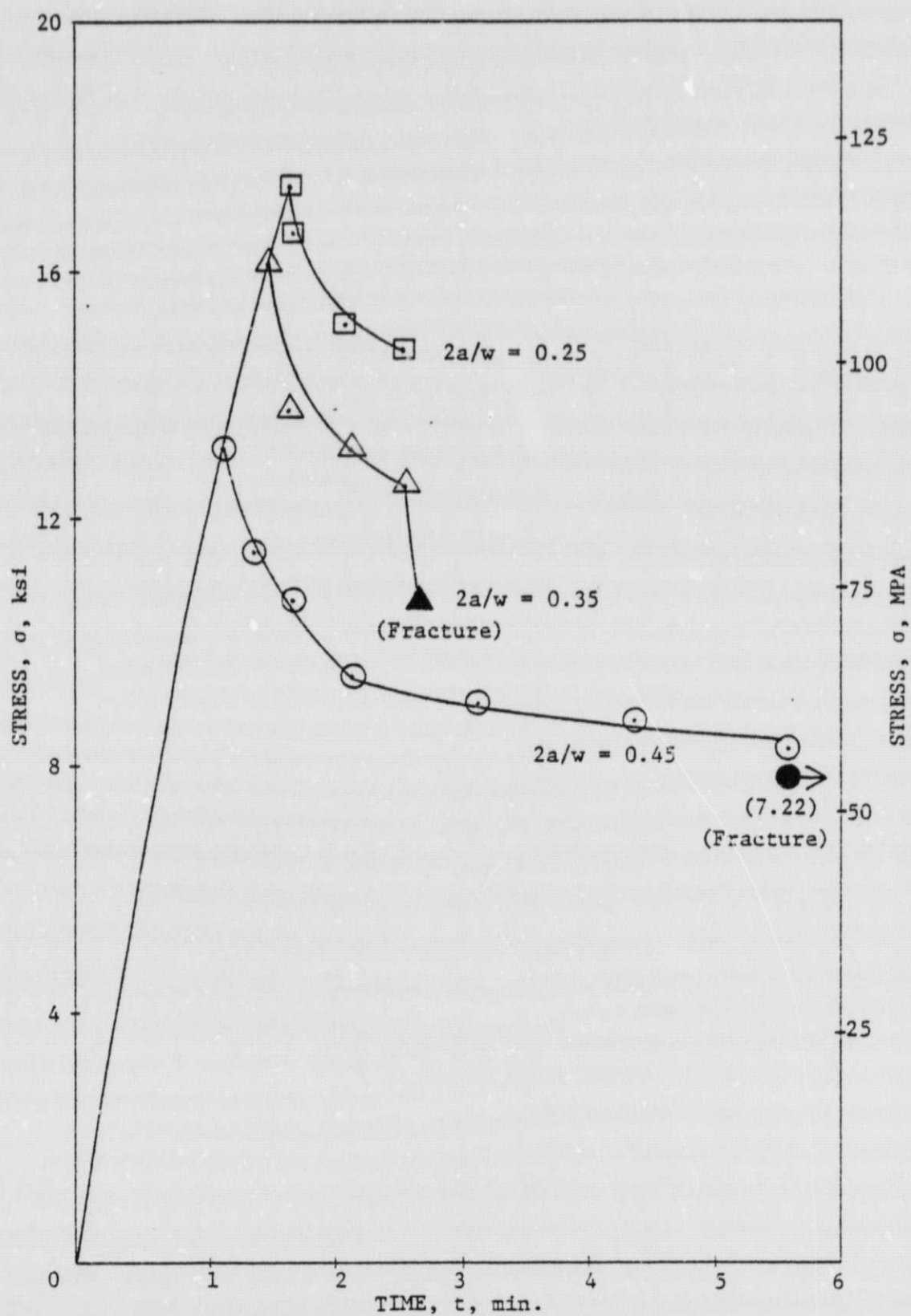
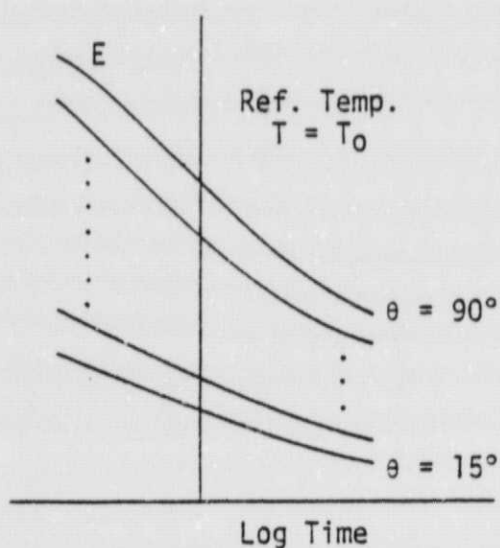
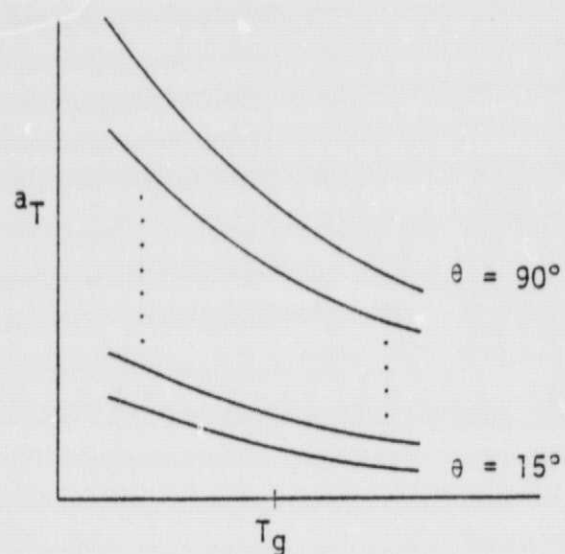


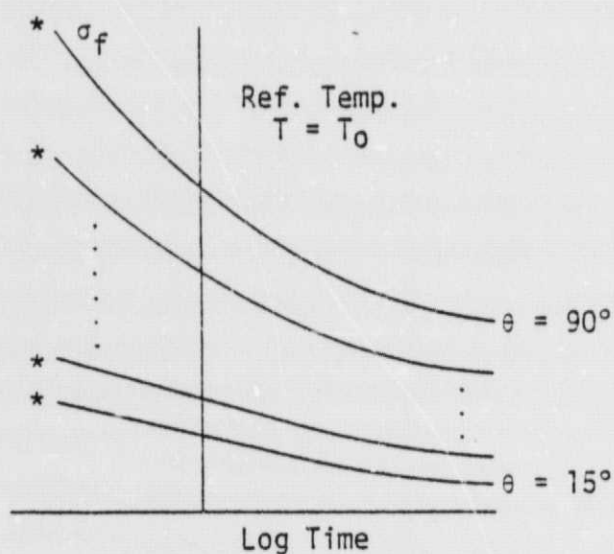
Fig. 1. Delayed or Time Dependent Fracture of $[\pm 45^\circ]_{4s}$ T300/934 G/E with Circular Holes.



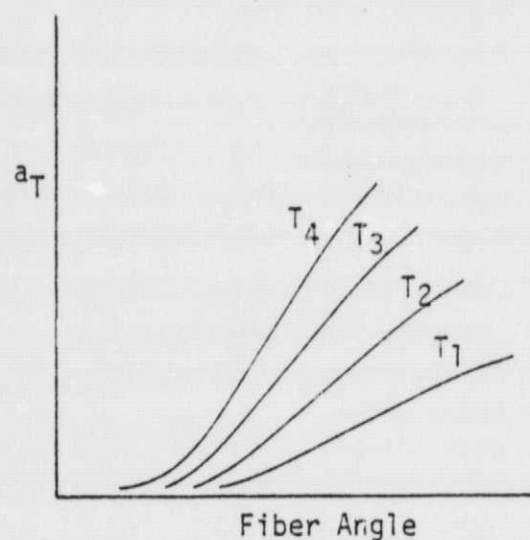
a) Measured Tensile Modulus Master Curves vs. Fiber Angle for Unidirectional Laminate.



b) Measured Shift Function vs. Fiber Angle for Unidirectional Laminate.



c) Tensile Strength Master Curve Assumed to Have Same Character and Shift Function as Modulus Master Curve (*Measured Data).



d) Formal Shift Function vs. Fiber Angle and Temperature Relation Analogous to WLF Equation Developed from b).

Fig. 2. Proposed Method to Determine Tensile Strength Master Curve and WLF Type Shift Function Equation.

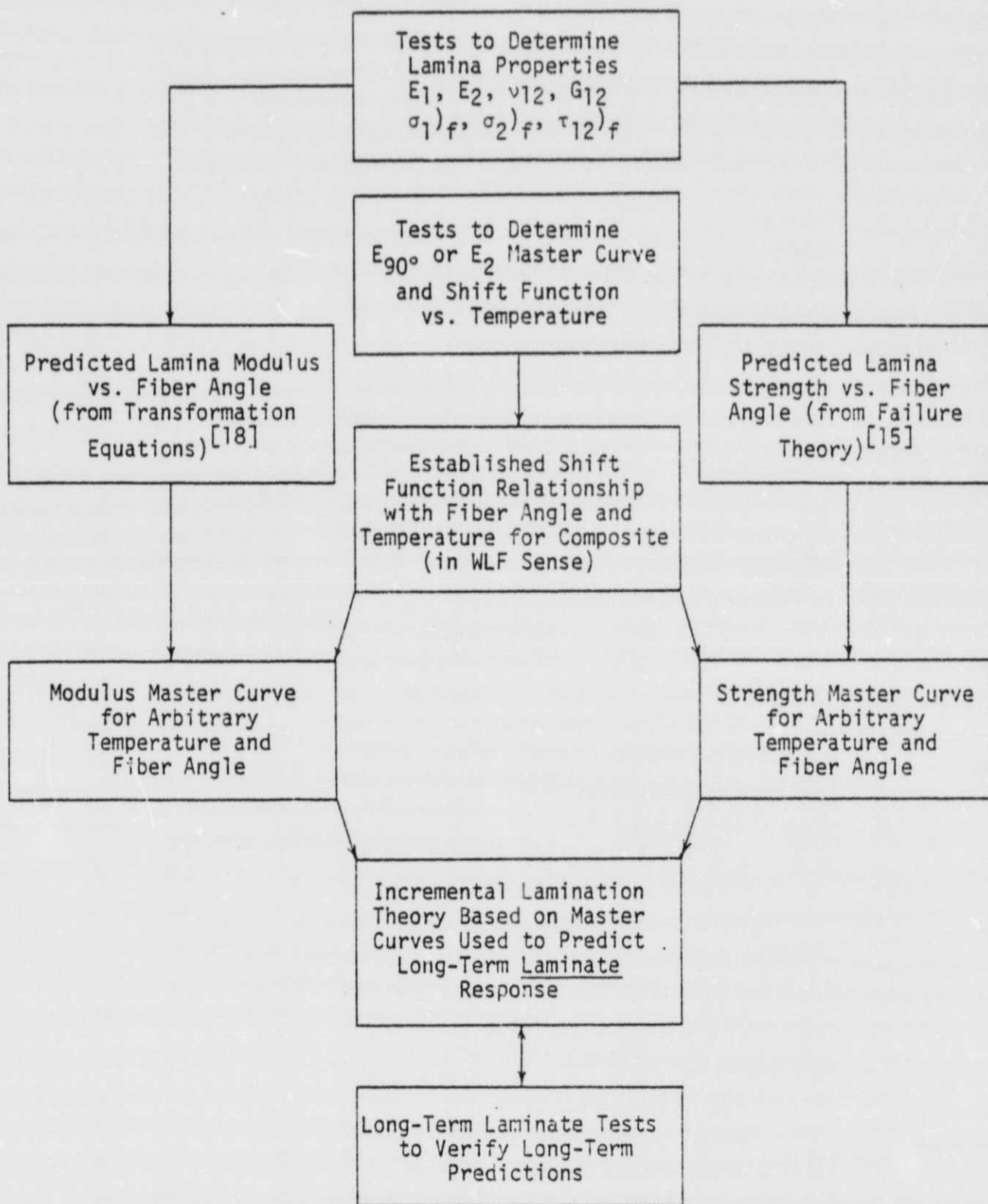


Fig. 3. Flow Chart for Proposed Laminate Accelerated Characterization and Failure Predictions Procedures.

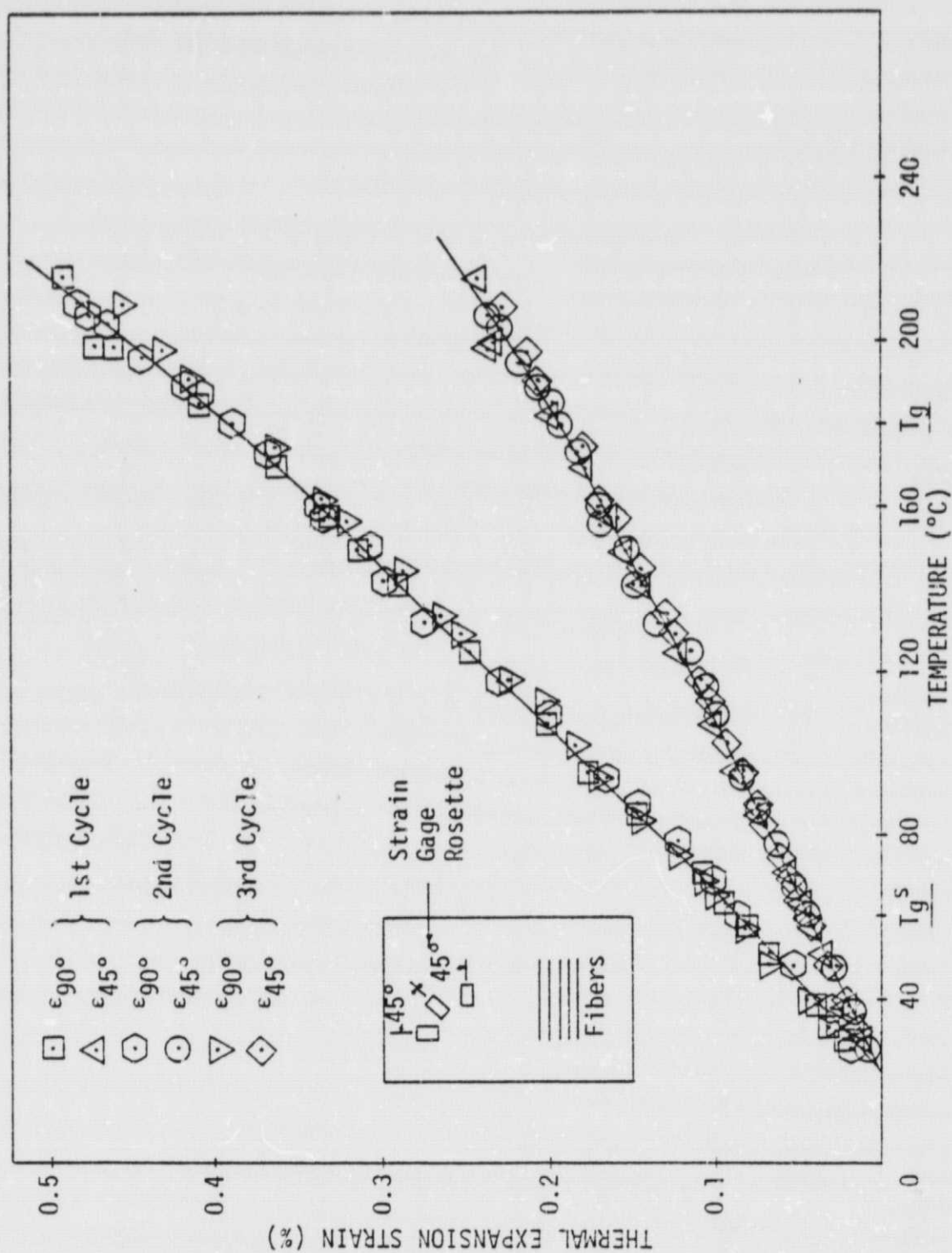


Fig. 4. Thermal Expansion of $[90^\circ]_8$ T300/934 G/E Laminate with Glass-Transition Temperatures Indicated.

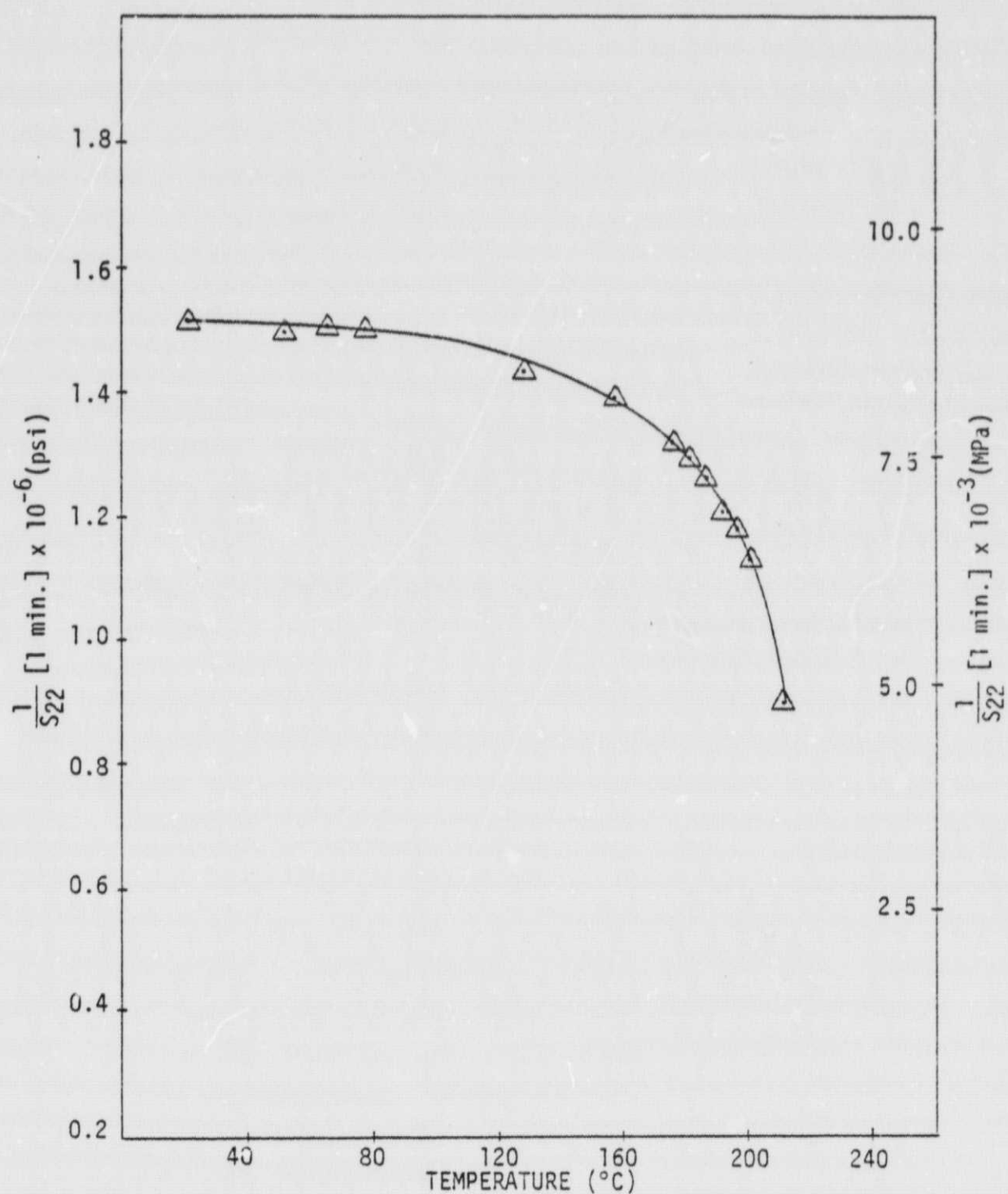


Fig. 5. One Minute Axial Creep Reciprocal of Compliance Showing Temperature Dependence for $[90^{\circ}]_{8s}$ T300/934 G/E Laminate.

- △ 20°C, 60°C, 100°C, 145°C, 180°C, 205°C
- 30°C, 65°C, 110°C, 155°C, 185°C, 210°C
- 40°C, 70°C, 120°C, 160°C, 190°C
- ▽ 50°C, 76°C, 127°C, 165°C, 195°C
- ◇ 55°C, 85°C, 135°C, 175°C, 200°C

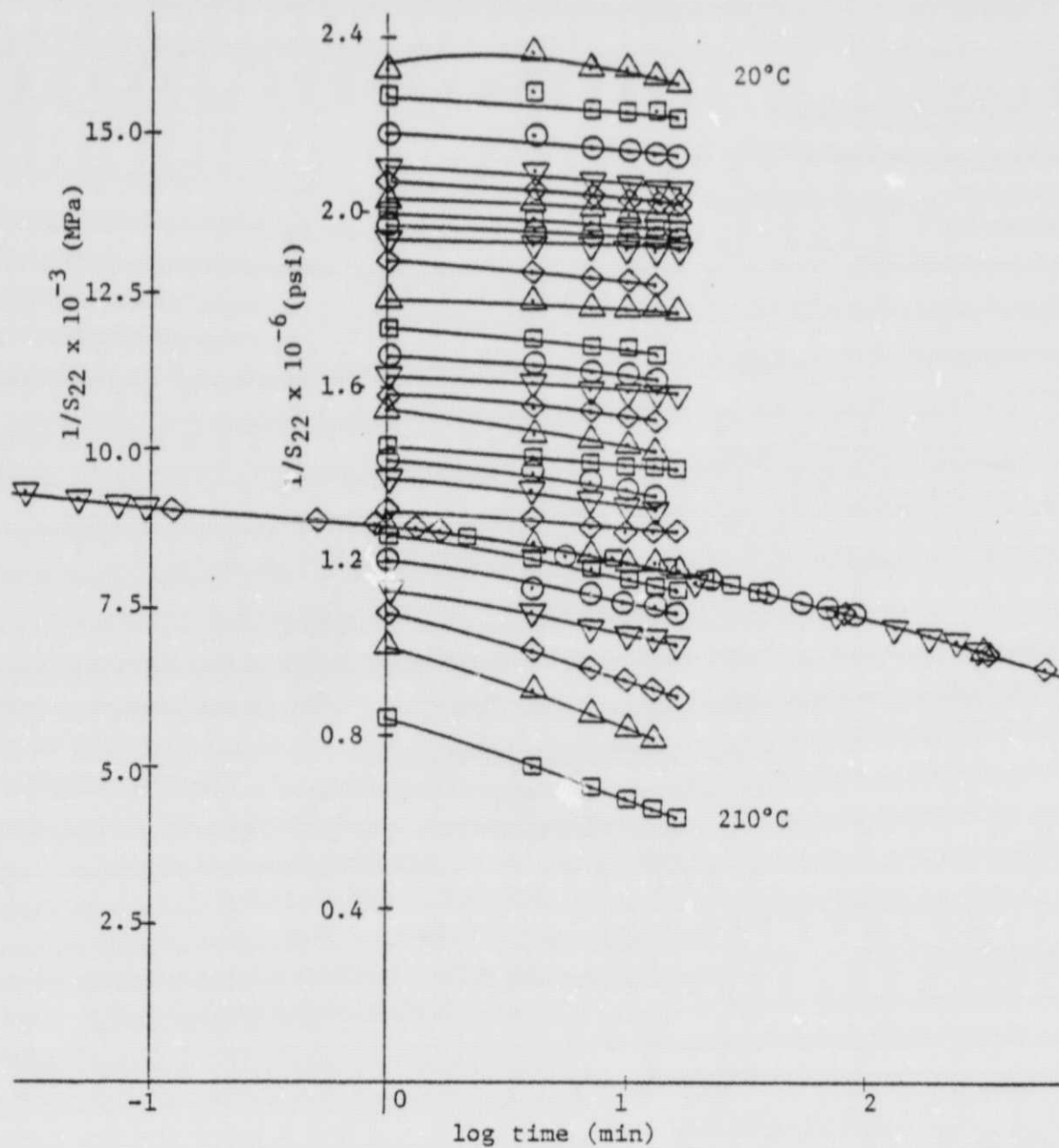


Fig. 6. Reduced Reciprocal of Compliance, $1/S_{22}$, and Portion of 180°C Master Curve for $[90^\circ]_{8S}$ T300/934 Graphite/Epoxy Laminate.

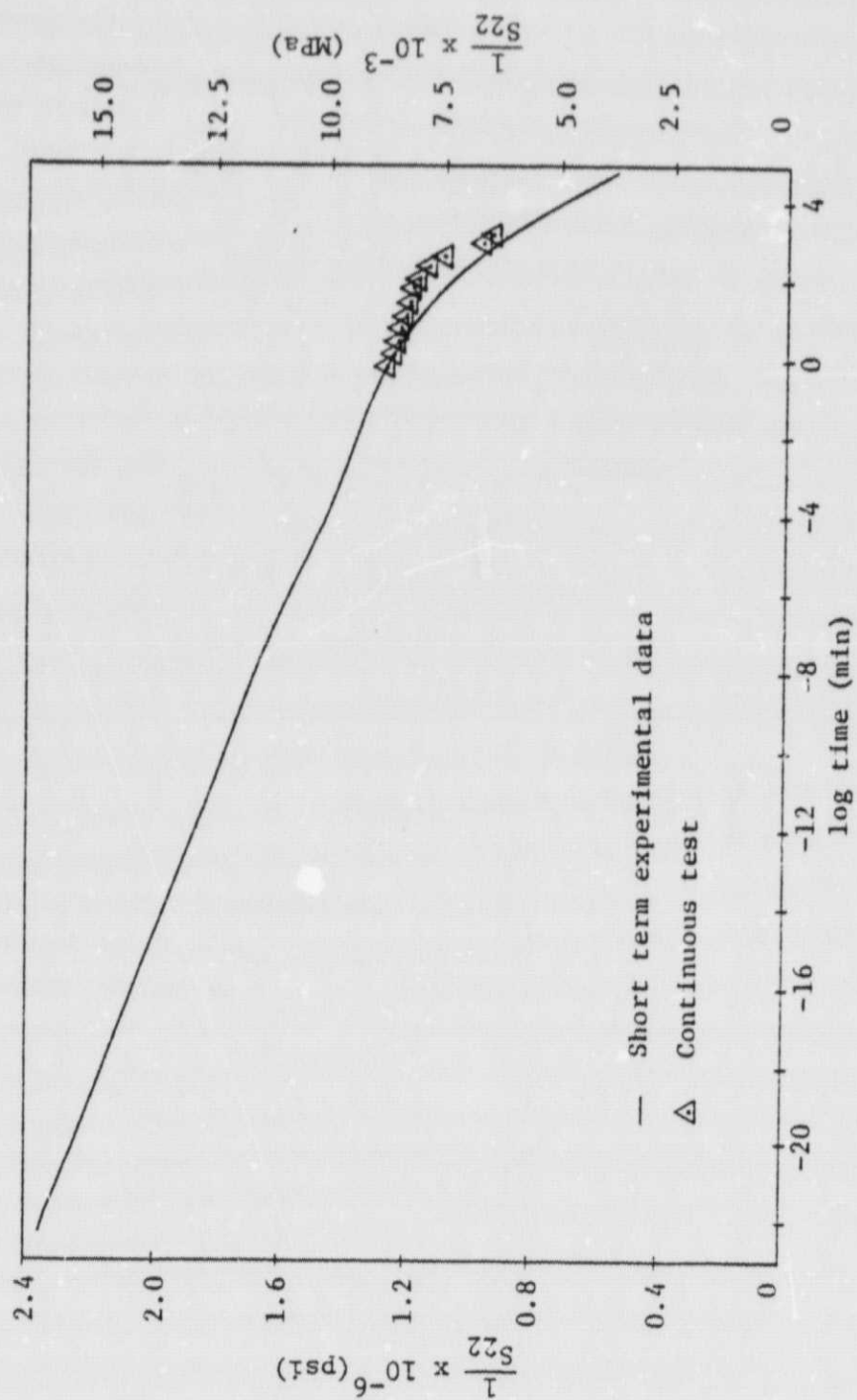


Fig. 7. Master Curve of the Reciprocal of Reduced Compliance, $\frac{1}{S_{22}}$ of $[90^\circ]_8$ s Laminate at 180°C .

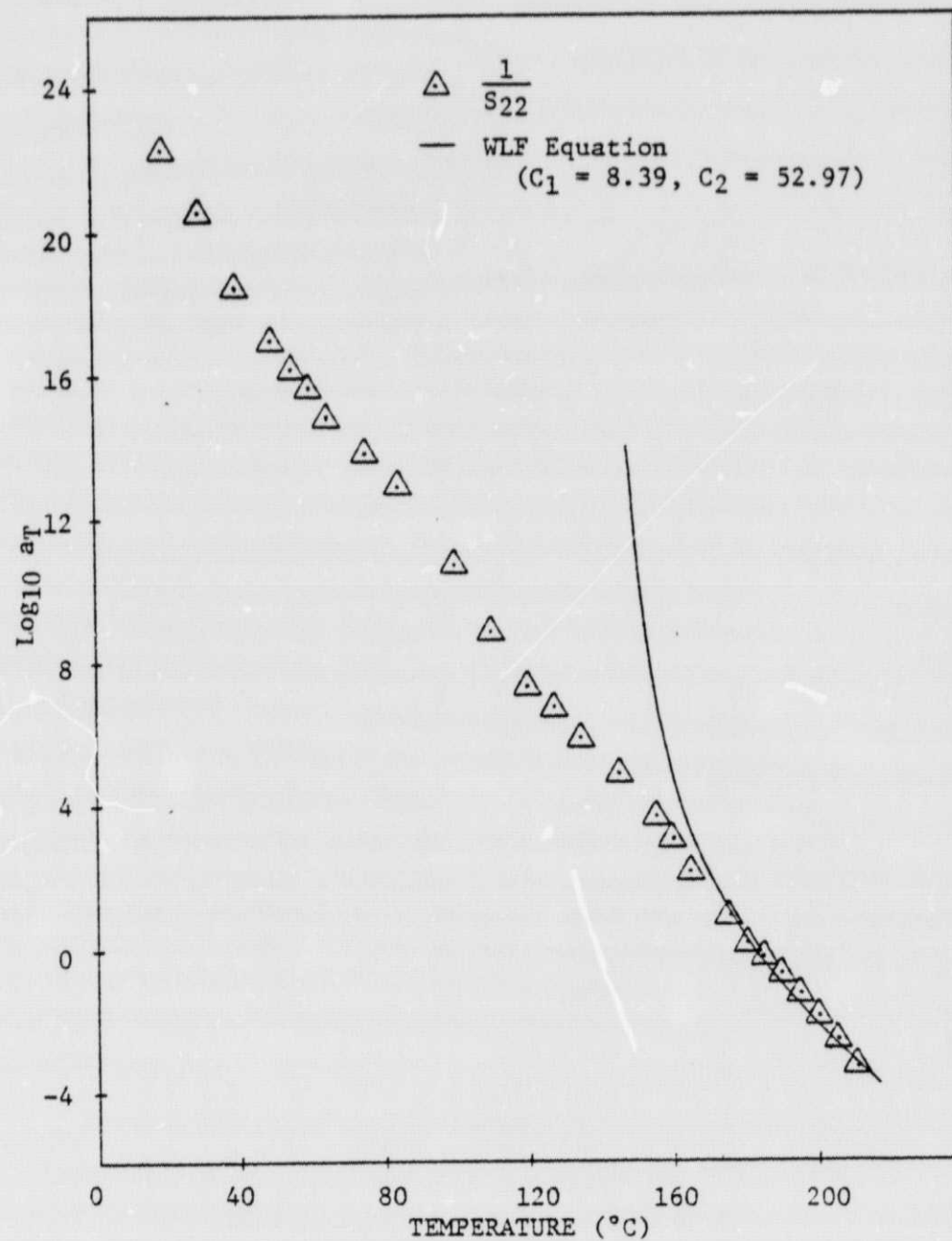


Fig. 8. Shift Factors, $\text{Log } a_T$, Versus Temperature.

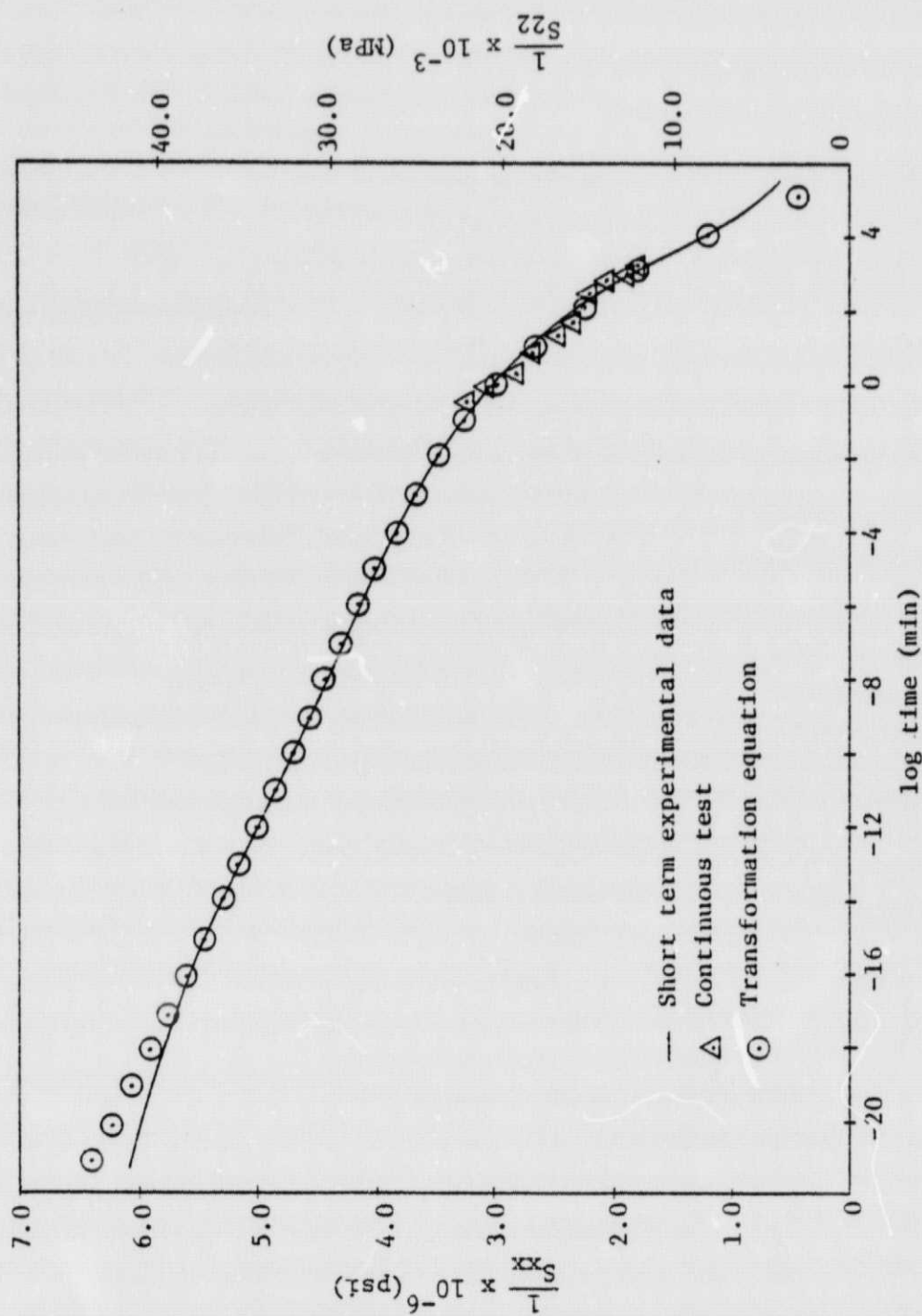


Fig. 9. Master Curve of the Reciprocal of Reduced Compliance, $\frac{1}{S_{xx}}$, of $[30^\circ]_{8s}$ Laminate at 180°C .

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